Characterization of Waterworks Waste for Use in Soil-Cement Bricks

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Abstract

In Brazil, the municipal waterworks generates huge amounts of sludge every year, and a critical issue is to find an adequate use for this waste material. The aim of this study is to determine the chemical, physical, and mineralogical characteristics of a waterworks waste sample from south-eastern Brazil, as well as to investigate its use in the production of soil-cement bricks. The waste sample was analyzed regarding to chemical composition, X-ray diffraction, particle size analysis, morphology, density, organic matter, soluble salts, cation exchange capacity, and pollution potential (leaching and solubilization tests). Soil-cement mixtures containing up to 5 wt.% of waterworks waste as partial replacement of soil were prepared, pressed, and cured for 28 days. The effects of the waste incorporation on physical properties such as compressive strength and water absorption have been determined. It was found that the waterworks waste is a material with high plasticity that is mainly composed of silica, kaolinite, gibbsite, and goethite. The results also showed that the waterworks waste has the potential to be used as an alternative raw material in the production of soil-cement bricks.

Keywords

Waterworks Waste; Characterization; Soil-Cement Bricks; Recycling

Introduction

The municipal waterworks are industrial units used to chemically and physically treat raw-fresh water mainly for human consumption. In general, the water treatment operation involves the following steps: i) pre-settlement of the raw water; ii) coagulation/ flocculation; iii) decantation; iv) settlement; v) filtration; vi) chlorination; and vii) pH control [Richter, 1991; Davis and Cornwell, 1998; Bernardo and Dantas, 2005]. As a result of these processes, a huge amount of waste in the form of sludge is produced worldwide.

The waterworks sludge after drying is considered to be a non-biodegradable waste material. This waste material has been mainly disposed near hydric resources and landfill sites, especially in developing countries. However, these methods of disposal generate significant environmental impacts and are not economically viable [Rodrigues, 2012]. In addition, the developing countries are now increasing their regulatory control on environmental problems, such as those relating to solid waste disposal. Thus, there is high interest in the development of technological alternatives for the reuse of this solid waste material.

Waterworks waste can be considered as a solid waste material rich in clay minerals, silt, and sand [Oliveira, 2004]. This means that the waterworks waste has the potential to be used as alternative ceramic raw material. On the other hand, the ceramic industry uses huge amounts of natural raw materials, mainly for the manufacture of building products. However, the ceramic industry is currently facing scarcity of good quality raw materials in locations close to the plants. In general, the waste materials show chemical, mineralogical, and physical characteristics compatible with the raw materials traditionally used in the ceramic industry [Silva and Holanda, 2009]. Nowadays, the use of industrial waste as an alternative raw material has attracted great attention worldwide [Badiee et al., 2008; Pinheiro and Holanda, 2009; Faria et al., 2012]. The waterworks waste has been studied as a possible new additive to clay-based products [Oliveira, 2004; Santos et al., 2001; Teixeira et al., 2004; Oliveira et al., 2006; Oliveira et al., 2007], exhibiting promising results. The reuse of waterworks waste for obtaining soil-cement bricks has also been recently suggested [Porras et al., 2008; Silva, 2009].

In the context of recycling, the purpose of this study was to investigate the characteristics and possible use of waterworks waste to produce soil-cement bricks. Despite the ceramic industry is highly promising for the final disposal of solid waste materials, little attention has been given to the recycling of waterworks waste in soil-cement bricks [Rodrigues, 2012].

Materials and Methods

The waste sludge sample used in the experimental

study was collected from a waterworks located in south-eastern Brazil (Campos dos Goytacazes-RJ). After drying at 110ºC for 24 hours, the waterworks waste sample takes the form of a fine clay-like powder.

Mineralogical analysis was performed by X-ray diffraction using monochromatic Cu-Kα radiation at a scanning speed of 1.5º (2θ)/min in a conventional diffractometer (XRD– 7000, Shimadzu). The crystalline phases were identified by comparing the intensities and positions of the Bragg peaks with those listed in the JCPDS-ICCD cards.

The chemical composition of the waste sample was determined by using energy-dispersive X-ray spectrometer (EDX700, Shimadzu). The loss on ignition (LOI) was determined according to the following equation:

$$LOI = (Wd-Wc)/Wd \times 100 \quad (1)$$

in which Wd is the weight of a sample dried at 110ºC, and Wc is the weight of the sample calcined at 1000ºC for 2 hours.

The particle size analysis of the waste powder was determined by a combination of sieving and sedimentation procedures, according to NBR 7181 [ABNT, 1984a]. The morphology of the powder particles was observed by scanning electron microscopy (SEM SSX-550, Shimadzu). The plasticity of the waste sample was determined by the Atterberg method according to the NBR 6459 [ABNT, 1984b] and NBR 7180 [ABNT, 1984c] standardized procedures. Other relevant characteristics such as organic matter content, pH, cation exchange capacity, and soluble salts contents were also determined.

DTA/TG experiments were carried out on the waterworks waste sample using a thermal analyzer (DTG–60H, Shimadzu), under air atmosphere from room temperature up to 1000ºC at a heating rate of 10 ºC/min.

The pollution potential of the waterworks waste sample was determined by leaching (acetic acid buffer solution (0.5 M) at pH 5.0 for 18 hours) and solubilization tests in aqueous media, according to the NBR 10005 [ABNT, 2004a] and NBR 10006 [ABNT, 2004b] Brazilian standards. The concentrations of elements present in the leaching and solubilization extracts were determined. The concentrations obtained were compared to the maximum concentrations of elements predicted by the NBR 10004 [ABNT, 2004c] Brazilian standard.

A series of soil/cement/waterworks waste mixtures was prepared. In this study, the soil was partially replaced with up to 5 wt.% of waterworks waste. A traditional soil-cement body (soil: cement–10:1) was used as a reference. Each mixture is labeled as follow: LW0 contains 0 wt.% waste; LW1 contains 1.25 wt.% waste; LW2 contains 2.50 wt.% waste; and LW3 contains 5 wt.% waste. Commercial Portland cement and soil were used. The soil/cement/waterworks waste were moistened with water at 16 wt.% of the total mass.

Cylindrical specimens (ϕ = 37.1 mm) were prepared by uniaxial pressing at 18 MPa, and then cured by 28 days in a humid chamber (~ 95 % humidity at 24 ºC).

The water absorption values were determined from weight differences between the as-cured and water satured pieces (immersed in cold water for 24 h). The compressive strength of the test specimens was determined by using an universal testing machine at a loading rate of 1mm/mm according to the NBR 10836 standard [ABNT, 1994a].

Results and Discussion

The X-ray diffraction pattern of the waterworks waste sample is shown in Fig. 1. The sample exhibited peaks that are characteristics of kaolinite ($Al_2O_3.2SiO_2.2H_2O$), silica (SiO_2), gibbsite ($Al(OH)_3$), goethite ($Fe_2O_3.H_2O$), and traces of illite/mica, with predominance of kaolinite.

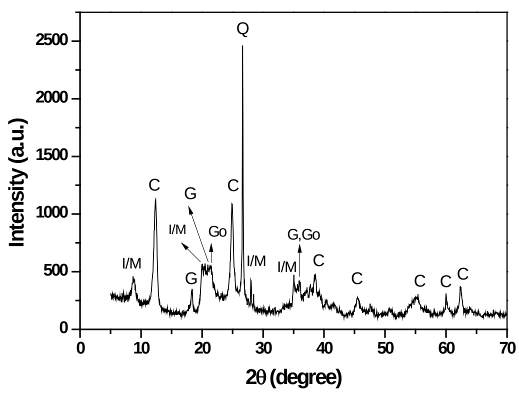

FIG. 1 X-RAY DIFFRACTION PATTERN OF THE WATERWORKS WASTE SAMPLE: C – KAOLINITE; Q – QUARTZ; G – GIBBSITE; GO – GOETHITE; I/M – ILLITE/MICA

The chemical composition of the waterworks waste sample is given in Table 1. In terms of chemical composition, the Al_2O_3 was the most abundant component, followed by SiO_2 and Fe_2O_3. This result is consistent with the X-ray diffraction data (Fig. 1). The alumina does not occur in its free form in the waterworks waste sample, being bounded to the clay minerals (kaolinite and illite/mica) and gibbsite. The

silica is present in the structure of clay minerals and free silica particles. The iron oxide is present in the structure of the goethite. Loss on ignition (LOI) implied in high weight loss of about 24.50 wt.%, is mainly attributed to the presence of clay minerals, hydroxides, and organic matter in the waste sample.

TABLE 1 CHEMICAL COMPOSITION OF THE WATERWORKS WASTE

Oxides	wt.%
SiO_2	29.59
Al_2O_3	31.18
Fe_2O_3	10.21
TiO_2	1.04
K_2O	1.27
SO_3	1.61
CaO	0.34
ZnO	0.02
MnO	0.14
Loss on ignition (1000 ºC)	24.50

Several important physical and chemical characteristics of the waterworks waste sample are given in Table 2. The waste sample presented true density of 2.50 g/cm^3, which reflected its mineralogical composition. The pH (in water) of the waste sample was 5.30, which can be considered as being of medium acidity. The sample presented a value of cation exchange capacity (CEC) of 6.89 meq/100 g within the CEC range of kaolinitic clays (3 – 15 meq/100 g) [Santos, 1989], corroborating the kaolinitic character of the investigated waterworks waste. It was also found that the waterworks waste presented low soluble salts contents (in water) and high organic matter content (25.86%). This result suggested that the organic matter significantly contributed to the high value of loss on ignition (Table 1).

TABLE 2 CHARACTERISTICS OF THE WATERWORKS WASTE

Characteristic	Value
True density, g/cm^3	2.50
pH (H_2O)	5.30
CEC, meq/100g	6.89
Organic matter, %	25.86
Soluble salts, ppm	
K	1.20
Ca	18.80
Na	1.50
Mg	6.60
Al	5.10
H + Al	40.80

The particle size distribution curve of the waterworks waste sample is shown in Fig. 2. The sample presented large particle size distribution varying between 1 and 600 μm. Most of the particles are in the silt size range ($2 \leq x < 63$ μm) with 62 %, followed by clay size range (< 2 μm) with 35 %, and sand size range (> 63 μm) with 3 %.

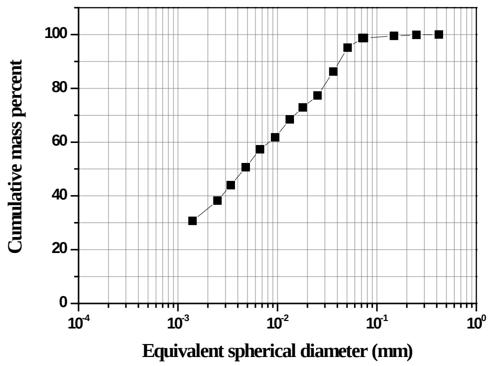

FIG. 2 PARTICLE SIZE DISTRIBUTION OF THE WATERWORKS WASTE SAMPLE

Morphological aspects of the waterworks waste sample observed by scanning electron microscopy are outlined in Fig. 3. It can be seen that the waste sample is rich in irregular-shaped particles which are probably kaolinite ones. In addition, a wide particle size range can be observed, in accordance with the particle size data (Fig. 2). Line spectrum for the waterworks waste determined using EDS is shown in Fig. 4, where Si, Fe, Al, C, O, and K were detected. These results are consistent with the chemical composition data (Table 1).

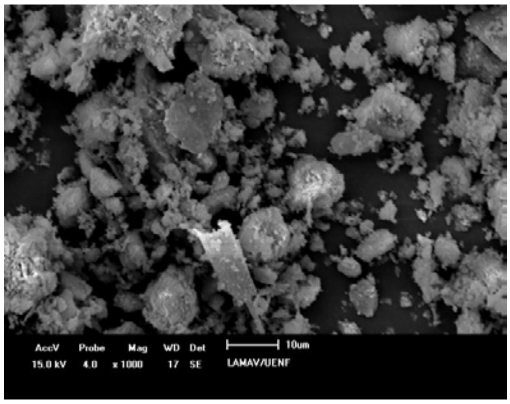

FIG. 3 MORPHOLOGY OF THE WATERWORKS WASTE PARTICLES

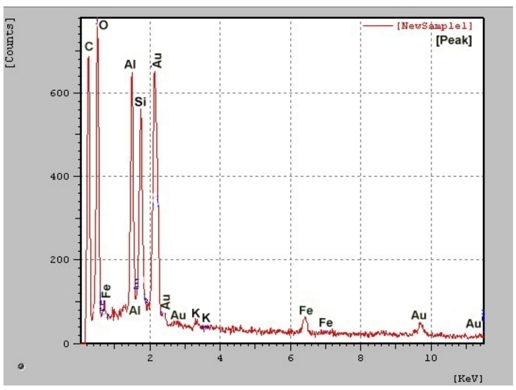

FIG. 4 EDS SPECTRUM FOR THE WATERWORKS WASTE PARTICLES

The plastic properties of the waste sample are presented in Table 3. In terms of plasticity, the waste

sample presented plastic limit (water of plasticity) of 32.8 %, liquid limit of 58.2 %, and plastic index of 25.6 %. Thus, the waterworks waste sample can be considered a clay-like material of high plasticity.

TABLE 3 PLASTIC PROPERTIES OF THE WATERWORKS WASTE

Property	Value
Liquid limit, %	58.2
Plastic limit, %	32.8
Plastic index, %	25.4

The thermal behavior (DTA and TG curves) of the waste sample is presented in Fig. 5. It can be seen that the investigated waterworks waste sample exhibited four thermal events at 57ºC, 265ºC, 318ºC, and 481ºC accompanied by an intense process of mass transfer. These events are associated with removal of adsorbed physically water (57ºC) on the surface of the mineral particles, removal of hydroxides crystallization water (265ºC), decomposition of organic matter and dehydration of sulphates used in the raw water treatment [Oliveira, 2004], and dehydroxylation of silicate lattice leading to the formation of metakaolin (481ºC), respectively. A small exothermic peak at 748ºC was observed likely related to the recrystallization of new phases, such as a Si-containing γ-Al2O3 or mullite [Gualtieri et al., 1995; Chen et al., 2000].

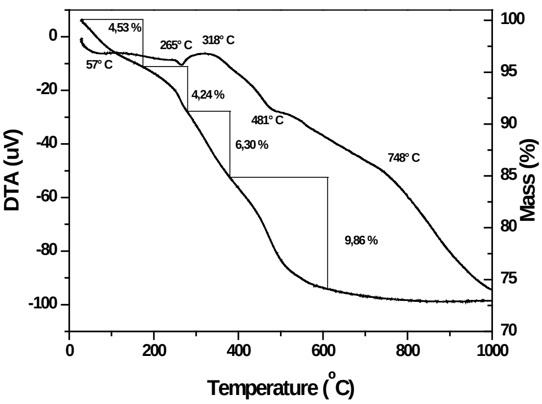

FIG. 5 DTA/TG CURVES FOR THE WATERWORKS WASTE SAMPLE

The ecotoxicity of waterworks waste was evaluated through leaching and solubilization tests, as shown in Tables 4 and 5. As observed in Table 4, the waterworks waste presented concentrations of Ag, As, Ba, Cd, Cr (total), Hg, and Pb below the maximum limits accepted by the NBR 10004 standard [ABNT, 2004c]. The solubilization results showed that in the waterworks waste sample, the aluminum (0.309 mg/L), iron (0.809 mg/L), manganese (19.481 mg/L), surfactants (0.79 mg/L), and sulphates (309 mg/L) concentrations exceeded the Brazilian solubilization limits [ABNT, 2004c]. Thus, the waterworks waste from south-eastern Brazil can be classified as a non-inert waste material (class IIA) according to NBR 10004 standard [ABNT, 2004c]. These results indicated that the disposal of the waterworks waste in water resources and the environment should be avoided.

TABLE 4 RESULTS OF THE LEACHING TEST FOR THE WATERWORKS WASTE

Elements	Waterworks waste(mg/L)	Brazilian limits(mg/L)
Ag	< 0.0130	5
As	0.0024	1
Ba	1.9900	70
Cd	< 0.0120	0.5
Cr (total)	< 0.0420	5
Hg	< 0.0001	0.1
Pb	< 0.0300	1

TABLE 5 RESULTS OF THE SOLUBILIZATION TEST FOR THE WATERWORKS WASTE

Elements	Waterworks waste(mg/L)	Brazilian limits (mg/L)
Ag	< 0.0130	0.05
Al	0.3090	0.2
As	0.0008	0.01
Ba	< 0.4100	0.7
Cd	< 0.002	0.005
Cr (total)	< 0.0100	0.05
Cu	0.0340	2
Fe	0.8090	0.3
Hg	< 0.0001	0.001
Mn	19.4810	0.1
Pb	< 0.006	0.01
Zn	< 0.0140	5
Nitrate	3.56	10
Sulphate	309	250
Surfactants	0.79	0.5
Hardness(total)	58.58	-

In this study, soil-cement bricks compositions were formulated using waterworks waste as a partial replacement for sandy soil. The quality of the soli-cement bricks after curing for 28 days was determined on the basis of compressive strength and water absorption.

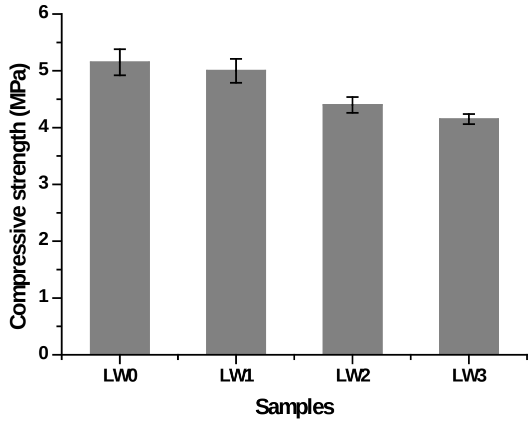

FIG. 6 COMPRESSIVE STRENGTH OF THE SOIL-CEMENT BRICKS

The compressive strength of the soil-cement bricks is shown in Fig. 6. As it can be observed that the

compressive strength of the bricks presented only slight decrement with waste addition. The values of compressive strength are in accordance with the limit values of the Brazilian specification ($\sigma \geq 2.0$ MPa) used for industrial production of soil-cement bricks.

Fig. 7 shows the level of open porosity of the soil-cement bricks evaluated by the water absorption. It can be observed that the waterworks waste containing bricks showed higher water absorption values than the reference bricks (LW0 sample). This is related mainly to the presence of high amount of clay minerals (kaolinite and illite/mica) and organic matter in the waterworks waste sample that tends to influence the cement hydration reactions [Rodrigues, 2012]. The specified value of water absorption for soil-cement bricks is equal to or less than 20% [ABNT, 1994b]. The above data suggested that additions of high amounts of waterworks waste into soil-cement bricks should be avoided, because it increases the open porosity of the pieces.

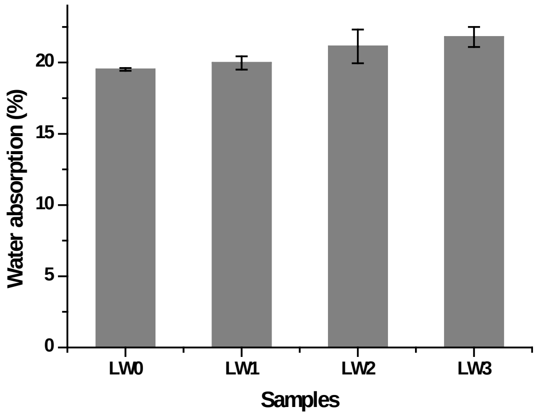

FIG. 7 WATER BSORPTION OF THE SOIL-CEMENT BRICKS

Conclusions

The following conclusions may be drawn from the experimental results and discussion.

- The waterworks waste characterization showed that material with high plasticity is rich in Al_2O_3, SiO_2, and Fe_2O_3, and organic matter. The results of X-ray diffraction showed that the major crystalline phases are kaolinite, silica, gibbsite, and goethite, with predominance of kaolinite.
- Leaching and solubilization tests indicated that, according to Brazilian standards, waterworks waste could be classified as class IIA waste material (non-inert).
- The limitations for incorporation of the waterworks waste into soil-cement bricks result from the increase of water absorption. Up to ~ 2.0 wt.% waterworks waste can be incorporated in soil-cement bricks compositions.
- The incorporation of waterworks waste in soil-cement bricks is an excellent alternative for material reuse and waste recycling practices.

ACKNOWLEDGMENT

The authors acknowledge the CNPq and FAPERJ for supporting this work, and also the Águas do Paraíba Company by the supply of the waterworks waste.

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